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THE APPLICABILITY OF CONTINUOUS SOLVENT EXTRACTION TO TUNG OIL

By

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INTRODUCTION

Solvent extraction, although widely used in Europe on practically every sort of oil-bearing seed, has been employed in the United States principally for extracting soybeans. Application of the process is expanding in the soybean industry, and it is being extended to other oil seeds. With the growth of the domestic tung industry, interest in the recovery of tung oil by solvent extraction is increasing. This report presents a résumé of the status of this method of extraction in regard to its applicability to tung.

There are in general two distinct types of solvent-extraction: batch and continuous extraction. In this report only continuous extraction is considered, although batch extraction probably could be applied to tung.

GENERAL

Solvent-Extraction Systems

In a complete solvent-extraction plant, various auxiliary units are required in addition to the principal unit, the extractor itself. There must be storage space for the raw materials, solvents, and finished product; facilities for the preparatory drying, flaking, and conditioning of the material; evaporators for the concentration of the miscella; a recovery system for the solvent distilled from miscella and removed from the extracted material, and strippers to remove the last traces of solvent from the oil.

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[1] Acknowledgment is made to L. F. Martin and R. S. McKinney of the Bureau of Agricultural and Industrial Chemistry for much assistance in the preparation of this report, and E. A. Gastrock, Engineering and Development Division, and A. E. Bailey, Girdler Corp., Louisville, Ky., for suggestions and additions to the information presented.

A number of continuous solvent-extraction systems are on the market which differ both as to the type of extractor and the design of the various auxiliary units. There are at least five different types in commercial use on soybeans in the United States and others are in the developmental stage. Still other types of extractors have been used commercially abroad. Sufficient data have not been published to prove the superiority of any particular type for the extraction of tung oil. Details of their construction and relative merits, therefore, will not be discussed in this report. Descriptions of the principal commercial types are given by Markley and Goss[2/] and by Bailey,[3/] and a list of firms which have developed or are developing complete solvent-extraction systems is given below.

Firms Which Have Developed or are Developing Complete Solvent-Extraction Systems

Allis-Chalmers Mfg. Co., 1941 Ristow Street, Milwaukee 1, Wisconsin

Blaw-Knox Co., P. O. Box 1198, Pittsburgh, Pennsylvania

Buffalo Foundry and Machinery Co., (Bollman system) 1575 Fillmore Avenue, Buffalo, New York

Detrex Corp., (Trichlorethylene system) 13005 Hillview Avenue, Detroit 27, Mich.

French Oil Mill Machinery Co., Piqua, Ohio

Read Machinery Co., (Kennedy system) York, Pennsylvania

Sieck & Drucker, Inc., (Hildebrandt system) 9 S. Clinton Street, Chicago, Ill.

Struthers-Wells Corp., (Hildebrandt system) Warren, Pennsylvania

An extensive list of manufacturers of extractors and auxiliary equipment is available in another mimeographed publication[4/] of the Bureau of Agricultural

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[2/] K. S. Markley and W. H. Goss. Soybean Chemistry and Technology. Chemical Publishing Company.

[3/] Alton E. Bailey. Industrial Oil and Fat Products. Interscience Publishers.

[4/] Mimeographed publication, AIC-98. United States Manufacturers of Equipment for Processing Cottonseed and Peanuts into Oils, Meals, and Byproducts.

and Industrial Chemistry.

The principal advantages of solvent extraction as applied to oil seeds are: (1) recovery of practically all the oil; (2) low labor costs per ton in large-scale processing installations, continuously operated, and (3) production of meal with a very low residual oil content. It is generally considered that, to be economical in operation a solvent-extraction plant should have a capacity of at least 50 tons of raw material per 24-hour day, corresponding to 100 tons of tung fruit per day.

Selection of a Suitable Solvent

To be suitable for use in extraction, a solvent must be inexpensive and available in quantity; its boiling point must be low enough to permit its ready removal from the oil and residual meal, and high enough to permit condensation and efficient recovery of the temperature of the available condenser water; and, preferably, the solvent should be nonhazardous.

In general, the choice of a solvent for the most economical operation of an extraction process should depend upon several factors. A higher priced solvent may prove to be economical if it has greater effectiveness in removing oil from a given type of meal, thus increasing the capacity of the extractor and permitting operation with a smaller volume per ton of material extracted. As a result, less handling and evaporation would be required and operating costs, as well as solvent losses, would be reduced. Solvents having low specific heat and low latent heat of evaporation are preferable to solvents of approximately the same specific gravity but with higher specific and latent heats, provided the volume required per ton and solvent losses are comparable. The loss of solvent per ton is a more important item than evaporation, overhead power, labor, and other operating costs in present solvent-extraction processes.

The low cost primarily, with the secondary consideration of recoverability, has led to the use of petroleum solvents in nearly all plants now in operation. At current prices other solvents would have to possess a combination of advantages to compete with the petroleum solvents. The loss of 10 gallons of the cheaper petroleum solvents per ton of material extracted would not be more expensive, for instance, than the loss of one gallon of trichlorethylene per ton. The use of the cheaper solvents would also be desirable even in modern solvent extraction plants where the loss can be kept below one gallon per ton.

The chlorinated solvents have the advantage of being nonflammable and nonexplosive, and their use thus permits a considerable saving in insurance rates. Of these, trichlorethylene is available with stabilizers and is used commercially. Some other chlorinated solvents, such as carbon tetrachloride, are unstable, corrosive, and toxic. Trichlorethylene is claimed to be a more effective solvent for extracting oil seeds than are the petroleum hydrocarbons. Its use would thus mean an increase in the operating capacity of the extractor and a decrease in the volume of solvent required per ton of material extracted. Its specific and latent heats are quite low, but its specific gravity is twice that of hexane or heptane, so that more heat would be required to evaporate a given volume than would be required for a petroleum solvent. The specific gravity of trichlorethylene is higher than that of the meal to be extracted; hence, the meal would float on its surface and its use is not possible in those types of extractors which depend upon the sinking of the material in the solvent. There are systems on the market which operate effectively with this solvent.

Important properties of technical and commercial grades of solvents that might be considered for tung oil extraction are given in Table I, together with prices.[5]

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[5] Oil, Paint, and Drug Reporter, June 25, 1945.

TABLE I

PROPERTIES AND PRICES OF VARIOUS SOLVENTS

Solvent	B.P. or Range ° F.	Specific Gravity (Density) 68° F.	Specific Heat 68° F. B.T.U./lb.	Latent Heat B.T.U./lb.	B.T.U. to heat from 70° F. and evap. at boiling point per gallon	Price/gal. tanks[1]
V. M. P. naphtha	--	--	[2]	[2]	--	$0.065
Solvent naphtha	--	--[2]	[2]	[2]	--	0.075
Solvent hexane	140-158°	0.660[2]	0.94[2], [3]	143 [2]	1190	0.105-0.11
Solvent heptane	170-239°	0.684[2]	0.88 [2]	137 [2]	1460	0.105
Benzol, tech.	176°	0.879	0.73	170	1810	0.15
Isopropyl ether	153°	0.73	--	--	--	0.27
Alcohol, ethyl	172°	0.801	--	--	--	0.51
Alcohol, ethyl abs.	173°	0.789	1.04[3]	367	3120	0.54
Ethyl acetate	171°	0.90	0.84	153	1785	0.84-0.93[4]
Trichlorethylene	190°	1.455	0.40	103	1830	1.03 [4]
Chloroform	143°	1.50	0.42	106	1710	2.50-3.50[4]

[1] Prices from Oil, Paint, and Drug Reporter, June 25, 1945.

[2] Values given are for pure n-hexane and n-heptane; values for naphthas are not available but would be of same order.

[3] Specific heat over range 32° - 122° F. for n-hexane; at 77° F. for absolute alcohol.

[4] Prices on trichlorethylene and chloroform are for drums, carload lots.

SOLVENT EXTRACTION OF TUNG OIL

Review of Literature

As a basis for discussion of the status of solvent extraction as a suitable process for recovery of tung oil, pertinent publications to date of the Bureau of Agricultural and Industrial Chemistry are reviewed below.

Freeman and McKinney[6] found that in laboratory extractions of tung oil it was necessary to regrind the samples after partial extraction to obtain complete extraction. Three commercial firms were able to grind the meal so that 98-99% of the oil could be extracted in the laboratory without regrinding. In a batch pilot plant, because of channeling, lower extractions were obtained on meal than expected from laboratory extractions on the same material. Channeling was not encountered when extracting unground expeller cake; the efficiency of extraction was lower on ground than on unground expeller cake, probably because of channeling. Miscella was evaporated to about 30% solvent, and then stripped under vacuum. Oil extracted from expeller press cake was of somewhat lower quality than expeller oil, but it still met the minimum specifications of the American Society for Testing Materials. By layering the meal with gravel, extraction efficiencies as high as 98% were obtained. The statement is made that solvent-extracted oil tends to change to the beta-glyceride, but no evidence of this is presented. Rose, Freeman, and McKinney[7] carried out laboratory extractions in a glass extractor operating on the Soxhlet principle. Skellysolve F (b.p. 95° - 149° F.) was used as the solvent and the samples were extracted for about eight hours. Kernels ground in meat grinders could not be extracted below 8-10% oil without regrinding. The solvent-extracted oil, containing probably .5% solvent, solidified because of conversion to beta-glyceride after standing for two or three days.

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[6] Freeman, A. F., and McKinney, R. S. Tung Oil Extraction by a Solvent Process. Oil, Paint, and Drug Reporter, 140, 5 (1941).

[7] Rose, W. Gordon, Freeman, A. F., and McKinney, R. S. Solvent Extraction of Tung Oil. Ind. Eng. Chem., 34, 612-613 (1942).

This transformation could have been caused by light but hardly by traces of iodine or sulfur. The solid oil could be permanently liquefied by heating at 200° C. for 30 minutes, but this treatment so lowered the iodine number and the refractive index that the oil did not meet the minimum A.S.T.M. specifications. In at least one case the stripped oil deposited a sediment and had to be centrifuged to be clarified.

Freeman, Pack, and McKinney[8] investigated 33 solvents in the laboratory by two methods, in order to determine their relative merits for the extraction of tung. In the first method, samples of oil were extracted as in the usual laboratory procedures for analysis; that is, the solvent was boiled and the vapors condensed and percolated through the material. In the second method, the samples were extracted at room temperature, and the miscellas were filtered from the residual meal and evaporated and stripped under reduced pressure. All factors considered, the most satisfactory solvents were the medium boiling petroleum naphthas (b.p. 140° - 248° F.), and trichlorethylene.

This work does not bear out the general statement that solvent-extracted oil solidifies. In the first procedure using hot extraction, two of the 33 solvents gave a solid oil, while only one of the 33 solvents gave a solid oil when the extraction was done at room temperature. The two solvents in the first case were petroleum naphtha, but other similar petroleum naphthas did not give a solid oil under these conditions, nor did those naphthas which produced a solid oil in hot extraction produce a solid oil in extraction at room temperature. The only solid oil produced in the latter case was from ethylene dibromide. Also, every sample extracted at room temperature showed cloudiness or various amounts of precipitates except those samples extracted by isopropyl ether and trichlorethylene. This would indicate the necessity of filtering solvent-extracted oil after stripping. The oils extracted at room temperature with trichlorethylene remained entirely clear and liquid, while those from petroleum naphtha extractions did not.

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[8] Freeman, A. F., Pack, F. C. and McKinney, R. S. Solvents in Extraction of Tung Oil. Ind. Eng. Chem., 35, 1156-1159 (1943).

McKinney, Rose, and Kennedy[9] investigated the continuous process for solvent extraction of tung oil, using a Kennedy continuous countercurrent extractor. A commercial grade of normal hexane was used as the solvent. For the first experiments material was prepared on an attrition mill, but in later work the best results were obtained by reducing the kernels to a medium fine meal in a Wolf mill equipped with corrugated rolls (6" x 6" with 6 corrugations to the inch) and then passing this meal through smooth rolls to flake it. No preparation was necessary for the expeller press cake.

Extraction efficiencies of 99% - 99.5% were obtained in both meal and press cake by passing the meal twice through the extractor. When expeller cake was being extracted the concentration of the oil in miscella was higher than in the material being extracted. This was not true when meal was being extracted, and indicates that the oil in cake is more readily extracted, most of it probably adhering to the surface of the particles.

The oil from the first four experiments had a very high acidity (58.1 for Run No. 4) and solidified by changing to the beta-glyceride. Later runs were on fresher raw material and no trouble was encountered with solidification of the oil. Press cake oil had a greenish-brown color and was below A.S.T.M. standards for iodine value and Browne heat test. Most of the oils after stripping deposited voluminous precipitates, indicating the necessity for filtering solvent-extracted oil after it has been stripped. Difficulty was encountered in stripping completely even under vacuum unless live steam was injected into the lower part of the stripping column.

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[9] McKinney, R. S., Rose, W. Gordon, and Kennedy, Amgus B. Continuous Process of Solvent Extraction of Tung Oil. Ind. Eng. Chem. 36; 138-144 (1944).

Freeman, Pack, and McKinney[10] investigated the effect of moisture on grinding of tung kernels and solvent-extraction of meal. The most efficient extraction of tung meal in the laboratory using petroleum naphthas was found to occur between 6% and 9% moisture. The evidence indicates that it is the effect of the moisture on grinding efficiency that influences the extraction, rather than a direct effect of moisture content on extractability of the oil.

McKinney and Oglesbee[11] investigated variations in solvent-extracted tung oils from the solvents employed. Two commercial grades of each of the solvents, hexane, heptane, and trichlorethylene, were studied as extractants for tung oil. The extractions were carried out in the laboratory by hot percolation. The oils extracted by the hydrocarbons solidified after several months, at least partially, whether at room temperature or at 40° F. The oil from the trichlorethylene extractions remained liquid even at the lower temperature, but was of a darker color. The refractive index of this oil was higher than that of the other oils obtained and possibly indicates that trichlorethylene is a more selective solvent for the unsaturated glycerides than are the other solvents studied.

Two runs were made in a Kennedy extractor using commercial hexane and heptane fractions on tung meal. An extraction efficiency of 99.9% was obtained with hexane and 98.9% with heptane.

Comparison of Solvent Extraction of Tung Oil with Expeller Pressing

In recovering oils of approximately equal value solvent extraction is more profitable in comparison with mechanical pressing when applied to materials of lower oil content. This can be seen from a comparison of the two methods as applied to tung meal with an oil content of 40%, and to soybean meal with an oil content of 20%. Assume that the nonoil component remains constant in weight in both processes (decrease from loss of moisture would be small), that the residual meal from the expeller contains 5% oil, and that from solvent extraction contains .5% oil.

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[10] Freeman, A. F., Pack, F. C., and McKinney, R. S. Effect of Moisture on Grinding of Tung Kernels and Solvent Extraction of Meal. Proc. American Tung Oil Association, 1944, p. 43.

[11] McKinney, R. S., and Oglesbee, Ruby E. Some Variations of Solvent-Extracted Tung Oils Resulting from the Solvents Employed. Proc. American Tung Oil Association, 1945, p. 43.

Soybeans

Meal, 20% oil = 20/(1.00 - .20) = 25% oil based on oil free residue.

Press cake, 5% oil = 5/(1.00 - .05) = 5.3% oil based on oil free residue.

Efficiency = $\frac{25\% - 5.3\%}{25\%}$ x 100 = 78.8% for expelling.

Extracted cake, 0.5% oil = .5/(1.00 - .20) = .503% oil in solvent extracted cake.

Efficiency = $\frac{25\% - .503\%}{25\%}$ x 100 = 97.9% for solvent extraction.

Gain in pounds of oil per ton of soybeans extracted as compared to expelling = (97.9 - 78.8)% x 20% x 2000 = 76.4 lbs.

Tung

Meal, 40% oil = 40/(1.00 - .40) = 67% oil in meal on oil free basis.

Cake, same as for soybeans in both expelling and extraction.

Efficiency = $\frac{67\% - 5.3\%}{67\%}$ x 100 = 92.1% for expelling.

Efficiency = $\frac{67\% - .503\%}{67\%}$ x 100 = 99.3% for solvent extraction.

Gain in pounds of oil per ton of tung meal extracted as compared to expelling = (99.3 - 92.1)% x 40% x 2000 = 56.8 lbs.

While less gain is indicated for the higher oil content tung meal, it is nevertheless of interest to note that the 56.8 pounds of additional oil recovered by solvent extraction is equivalent to approximately 28.4 pounds per ton of tung fruit processed. Thus a mill processing 25 tons per day of tung fruit by expelling is losing 710 pounds per day of available oil, currently worth about $270. In an operating season of 150 days, the loss would amount to almost $40,000. Current prices cannot be expected to continue under normal market conditions, but at any profitable price for tung oil the gain by solvent extraction would be substantial. Another consideration in the case of tung is the fact that the expeller cake is used at present only as a fertilizer in which oil has no value, and thus any oil left in the cake is a total loss, whereas oil left in the expeller cake in other crops serves as stock feed.

The average percentage of oil contained in various seed meals are as follows: cottonseed, 25-40%; soybeans, about 18%; peanuts, about 50%, and tung, 40-50%. Thus, the percentage gain in recovery of oil by solvent extraction as compared to expelling would be greater for soybeans than for cottonseed, peanuts, or tung. However, the amount of seed meal to be handled per pound of oil recovered is greater in the case of the soybeans.

Solvent extraction of tung expeller cake after the bulk of the oil has been obtained by expelling has been proposed, and experimental extractions of the expeller cake have been made. While it probably would not be economical to combine solvent extraction of the expeller cake with the use of the high pressure expellers used in the United States, the use of special low pressure expellers in combination with solvent extraction of the expeller cake is widely used on other oil seeds in Europe,[12] and such a system might be feasible in the tung industry. The preparation of the tung seeds for solvent extraction is one of the greatest problems connected with the solvent extraction of tung, and this system would largely obviate that problem.

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[12] Klare S. Markley. Fats, Oils, Oil Seeds, and Related Industries in Germany and Northwestern Europe. PB-18302. Publication Board, U. S. Department of Commerce, 1946.

The oil has been economically recovered by a batch solvent-extraction process from the "foots" or press cake obtained in the filtration of expeller oil. In this case a small volume of material of high oil content is handled, and the nature of the material is such that relatively simple equipment suffices. At the present high price of oil it is not necessary to recover all of the inexpensive solvent used for profitable operation, but under normal price conditions even this operation might have to be conducted so as to recover solvent efficiently to be profitable.

Economics of Solvent Extraction of Tung Oil

The principal arguments at the present time for solvent extraction of tung are: the high current price of tung oil; the lack of value of oil left in the residual cake; lower operating and overhead costs than possible in expelling, provided enough material is available in a producing area to supply a plant of 100 tons or more daily capacity continously throughout a sufficiently long season.

Other advantages are those inherent in continuous large-scale operation which can be made semiautomatic and more accurately controlled. By selection of the proper solvent it should be possible to produce oil of a uniformly higher quality, and to modify or refine the oil in processing to reduce acidity and improve color. Residual cake would be subjected to less heating than in expeller pressing. Tung meal could be handled with as low a shell content as could be attained by hulling, so that there would be a greater possibility of using solvent-extracted cake as cattle feed, or for plastic and other protein byproducts.

Disadvantages to be considered are the larger capital requirement and initial cost of a solvent-extraction plant large enough for economical operation. While labor requirements in man-hours would be decreased, expert technical supervision would be necessary. Additional operations would be required in the proper preparation of material for extraction. The plant would have to be assured of a continuous supply of at least 100 tons of tung fruit per day for a sufficiently long season of operation, as shut-down would be more costly than in expeller operation. It would not be feasible to maintain such a plant on a seasonal basis. During the off-season it might be possible to operate on peanuts, soybeans, cottonseed, and probably other oil seeds from Central and South America. When the change to edible oils is made, extreme precautions would be necessary to prevent contamination with tung oil, or toxic constituents of tung meal.

The fact that the drying properties of tung oil, because of the conjugated double bonds of eleostearin, differ from those of other oils which have been solvent extracted must be considered. The general opinion is that in evaporating miscella and stripping residual solvent from tung oil, more care and lower temperatures would be necessary to avoid oxidation. However, tung oil extracted from expeller oil foots has been stripped of solvent at atmospheric pressure on a commercial scale and yielded oil of an excellent quality. If lower temperatures were essential to produce highest quality oil, vacuum evaporation and stripping could be employed.

A more important consideration is the tendency of some solvent extracted tung oils to solidify because of the formation of beta-eleostearin. In order to render the oil permanently liquid again it must be heated to 200° C. for 30 minutes. This heating injures the oil so much that it does not meet the A.S.T.M. specifications as regards iodine number and refractive index. The factors causing the formation of the beta-glyceride in solvent-extracted oil have not been determined. This tendency, which appears to be due to certain grades or types of solvents, may be due to traces of impurities or to conditions under which miscella and oil are evaporated and stripped. More experimental work is required to determine the exact causes of this transformation to the solid glyceride, and to establish conditions for its prevention.

Further Experimental Work Needed

Only in a modern efficient continuous countercurrent process of large capacity can the advantages and economies of solvent-extraction be fully realized.

The type of extractor to be used cannot be indicated, as this will depend upon the proper preparation of material and the selection of a solvent, but the design selected should give maximum recovery of oil. A rinsing solvent in a countercurrent system is essential to effect extraction to the lowest possible residual oil content, while at the same time building up the miscella to a high concentration and permitting operation with the minimum amount of solvent. Auxiliary equipment should be designed to effect as complete recovery of the solvent as possible. More complete removal of shell may be necessary as objectionable color or impaired quality may result from substances extracted from shell by the solvent. Preparation in a suitable form to minimize difficulty due to "fines" may also vary with the process and solvent used.

Studies which still need to be undertaken and completed before an all-solvent plant is erected include the following:

1. Methods of flaking kernels.

2. Beta-Glyceride formation in solvent-extracted oil.

3. Comparison of the qualities of solvent-extracted oil with expeller oil.

4. Comparison of the oils extracted by various solvents, especially of those extracted by hexane, heptane, and trichlorethylene.

5. Stripping of miscella without injury to the oil, and proper design of stripper.

6. Necessity of filtering oil after stripping, or means of preventing formation of precipitates.

Filtering stripped oil seems to be a step that has been omitted from the proposed processes for solvent-extraction of tung oil, but scattered through the published information there is considerable evidence that a precipitate forms in stripped tung oil from solvent extraction and that it would probably be necessary to filter such oil after it had cooled. It has been the experience in solvent extraction of "foots" on an industrial scale that the stripped tung oil deposited a rather bulky white precipitate and required filtration. This precipitate is probably a phospholipid on the order of lecithin, which is hydrated by steam introduced during stripping.

Tentative Procedure for Tung Oil Extraction

The work reported in the preceding review of literature shows that tung oil can be solvent-extracted with an efficiency of about 99 percent from either meal or press cake. The best procedure for tung, based on this work and on the experience with solvent-extraction in other vegetable oil industries seems to be as follows:

1. Use continuous countercurrent extraction. Only the Kennedy process has actually been tried out on both tung meal and press cake, but there are several other systems on the market which may be applicable.

2. Prepare material by cracking in corrugated rolls and then flaking between smooth rolls. It has been found at the Southern Regional Research Laboratory that peanuts flake best with a certain percentage of moisture which is critical within rather narrow limits.

3. Use commercial hexane as a solvent. Although trichlorethylene is a possibility, it cannot be used with the Kennedy extractor because of its high specific gravity.

4. Strip the oil under vacuum, or with live steam at atmospheric pressure.

5. Filter the oil after it has been stripped and cooled. With petroleum solvents and steam stripping, permanently clear oil cannot otherwise be obtained by methods tried thus far.

The only solvents about which enough is known to permit consideration for operation are the stright-chain hydrocarbon fractions which boil at 140° to 248° F. and the chlorinated hydrocarbons, dichloro- and trichloro-ethylene. Other solvents may become available at low prices as a result of developments during the war. Trichlorethylene is at present ten times as expensive as the cheaper hydrocarbons, but since it is not explosive and the loss per ton of material can be held to less than a gallon in modern extraction plants, its use might be competitive, particularly if it should prove to be a more effective solvent permitting operation at an increased capacity per extractor.

Summary

In this report the present status of solvent-extraction and factors to be considered in adopting this method for tung oil production have been presented as concisely as possible. Certain problems requiring further study have been indicated, which must be solved before the detailed steps of a complete solvent-extraction process for this application can be developed and recommended. Economics of the process as compared to present mechanical extraction are such as to warrant intensive study and development, and careful consideration of its advantages in localities where the extraction plant can be assured of 100 tons per day of fruit throughout the processing season.

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